Characterization of the dislocation structures–and twin boundaries in thin- and multilayers determined by X-ray line profile analysis

Gábor Csiszár

Supervisor:

Prof. Tamás Ungár

EÖTVÖS UNIVERSITY, BUDAPEST

GRADUATE SCHOOL OF PHYSICS

Material Science Program

Physics Ph.D program leader: Prof. Csikor Ferenc

Material Science Program leader: Prof. Lendvai János

EÖTVÖS UNIVERSITY, BUDAPEST

Department of Materials Physics

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Introduction

The topic of “*materials under extreme environments*” has received significant attention, because these materials are key building blocks for the next generation of energy technologies. They feature enhanced performance at extremes of mechanical stress, strain, temperature, pressure, corrosive environments, particle radiation flux, and electric or magnetic fields. These operating environments accelerate the aging process in materials reducing performance. Incremental changes in current structural materials may not satisfy the needs for future applications. Innovative basic research is required for setting the fundamentals of how materials behave in extreme environments. Controlling the matter-extreme environment interactions enable us to develop revolutionary new materials, extending their lifetimes, increasing efficiencies, providing novel capabilities and lowering costs.

Interest in nanocrystalline materials has provided superior multifunctional properties for advanced applications such as thin films and multilayers. In particular electrochemical deposition has become very attractive for dedicated fabrication of materials with microstructural features on the nanoscale. The internal structure and associated properties of electrochemically deposited films can be closely controlled by the applied process parameters. Nanoscale metallic multilayers are one of the basic key building blocks for the i.e.: fusion reactors due to the ultra-high strengths and radiation damage tolerance exhibited by these materials. Nanotwinned *fcc* thin films have also received significant attention due to the fabrication of *fcc* metals with nanotwinned structures using methods such as electro-deposition or sputter deposition. These as-grown nanotwinned structures exhibit unusual properties as compared to nanocrystalline metals. Typically, in the equi-axed grain nanocrystalline metals with high-angle grain boundaries the increased strength is accompanied by a loss in ductility, thermal stability and electrical conductivity. However, the nano-twinned metals such as Cu, exhibit very high tensile and fatigue strengths, with good ductility, thermal stability and electrical conductivity at room temperature.

X-ray diffraction peak profile analysis is a powerful alternative to TEM for describing the microstructure of these crystalline materials producing fundamental knowledge of the microstructure. X-ray diffraction peaks broaden when the crystal lattice becomes imperfect. The correlation between peak shape and specific lattice defects has been discussed in terms of the hierarchy of lattice defects. The appropriate size parameter, the average dislocation density with the prevailing Burgers vector structure can be generated by the method. Planar defects can also be characterized by the corresponding theoretical part of line profile analysis.

New scientific results

My doctoral dissertation consist of three different microstructural investigations where the major experimental method is X-ray line profile analysis. In the focus are three different thin films, namely Ni-phase nanocrystalline material, Cu/Nb multilayer and *de-twinned* Cu foils. The main results of my research are summarized briefly as follows.

I.

Nanocrystalline Ni thin films have been produced by direct current electrodeposition with different additives and current density in order to obtain <100>, <111> and <211> major fiber textures, respectively. The dislocation density, the Burgers vector population and the coherently scattering domain size-distribution is determined by high resolution X-ray diffraction line profile analysis. the CMWP procedure is used in a special manner to obtain the substructure parameters in the different coexisting texture components.

1) The dislocation density is found to vary in a wide range between 0.5 and 23×10^{15} m^{-2} versus both the different textures and the deposition conditions of the thin films.

2) The Burgers vector population is determined by analyzing the strain part of line broadening in terms of individual dislocation contrast factors. The matching of the measured and theoretical contrast factors provides the most probable Burgers vectors prevailing in the films. In the <100> and <111> textured films a considerable fraction of the Burgers vectors are parallel to the plane of the films. In the <211> textured films, however, the Burgers vectors are populated randomly.

3) The mechanical strength of the thin films is measured by Vickers hardness test and is correlated with the combined Taylor and Hall-Petch equation. Microstructural parameters determined by line-profile analysis provide the correlation between the microstructure and strengthening property.

4) The sub-structure parameters are correlated with deposition conditions. It is found that the largest current density and the largest amount of additives produce the highest dislocation density and the largest strength value.

II.

The dislocation density and the Burgers vector population is determined within the Cu and Nb layers in highly-textured sputter-deposited Cu-Nb multilayers by X-ray line profile analysis. I have characterized the dislocation populations in as-deposited film both as a function of (i) individual Cu and Nb layer thickness and (ii) total film thickness (with thinner films on substrates and thicker films self-supported). The key findings from this x-ray study of the as-deposited Cu-Nb sputter films are the followings:

1) In the Cu layers, the values of ρ are ρ=1.7 (±0.2) 10^{14} m^{-2}, irrespective of the layer thickness or the total thickness of the foils.

2) The dislocation density in the Nb layers increases slightly when the layer thickness decreases, but does not scale linearly.

3) The structure of the Burgers vector population does not depend on the layer thickness, or on the total thickness of the foils.

4) In both, the Cu and the Nb layers the overwhelming majority of the Burgers vectors of the edge dislocations are parallel to the foil planes. The dislocation character and the Burgers vector populations in the Cu layers suggest that a possible arrangement of the interface dislocations are arranged in a network of threefold symmetry.

5) If all the dislocations in the as-deposited films are contained in the interfaces, then increasing the layer thickness would result in a linear decrease in dislocation density. The fact that such a linear variation of dislocation density with layer thickness is not observed could be due to a combination of two factors: (i) the density of misfit dislocations in the interface plane gradually increases with layer thickness, and (ii) the density of dislocations within the layers decreases with decreasing layer thickness since dislocations in the layer interior but close to the interfaces are attracted to the interfaces.

6) The misfit dislocation spacing gradually decreases and asymptotically approaches the equilibrium value with increasing film thickness.

7) The strength of the foils is modeled in terms of the dislocation density and the Hall-Petch behavior. The strength of the foils decreases by about 20 % from 20 to 75 nm layer thickness, mainly because the layer-thickness increases.

III.

X-ray diffraction experiments have been performed on as-sputtered and room temperature rolled *de-twinned* Cu foils. The measured diffraction patterns are analyzed using the whole profile analysis approach to obtain average twin boundary densities (frequencies) and dislocation densities independently. The evolution of the average twin lamellae thickness and dislocation structure needed to revise the only descriptive well-known Hall-Petch relation to analyze more preciously the microstructural property of the material.

1) The whole peak profile analysis shows that the twin boundary density decreases by about 20 to 25 % after rolling changing the average distance of the twin lamellae of about 2-4 nm to 5-6 nm.

2) The dislocation density changes from $\rho \cong 4.4\times10^{14}$ m^{-2} to $\rho \cong 3\times10^{15}$ m^{-2} during rolling.

3) The increase in the flow-stress during rolling, despite the increase of d_{twin}, indicates that the dislocation density plays a major role. Twinning alone cannot be considered as the only reason of strengthening. The total value of flow stress is produced by the concomitant effect of twinning and dislocations.

4) The quantitative results of dislocation densities and twin boundary frequency are correlated with the strength of the thin foils. The Hall-Petch type weakening caused by detwinning is overcompensated by the Taylor type work hardening during rolling.

5) The α constant in the Taylor equation is found to be α=0.13. This relatively small value indicates, on the one hand, that the dislocations are in narrow dipole configurations, and on the other hand, it explains the good ductility despite the large strength of the foils. This features correlate well to the fact that dislocation cell structures do not form in *de-twinned* metals, a high density of dislocations can be stored at the twin boundaries that are strong obstacles to slip transmission.

Publications

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